ANALYTICAL RESOURCES CORE

COLORADO STATE UNIVERSITY

ARC MONTHLY BULLETIN

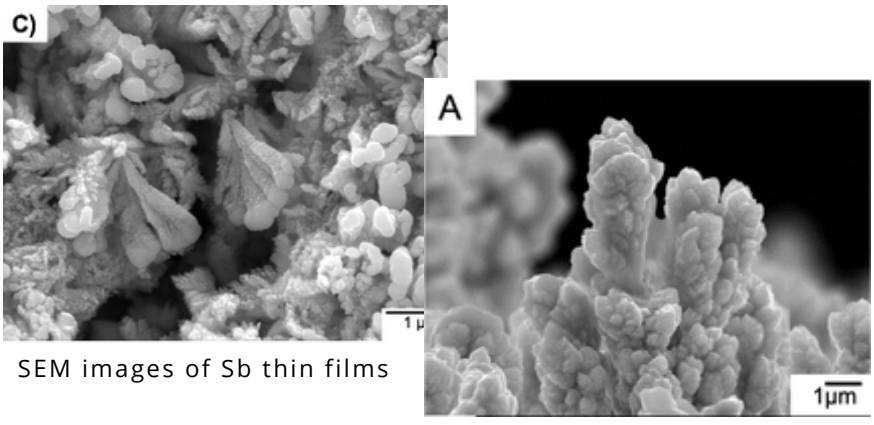
AUGUST 2023

Welcome to the ARC Bulletin, a monthly newsletter to keep you informed about the latest happenings in the ARC. Here you will find information about our team, job opportunities, equipment and facilities, upcoming seminars, and other exciting news!

RESEARCH SPOTLIGHT

This month's research spotlight features Dr. Kelly Nieto's recent publication in the Journal of Physical Chemistry **"Structural Control of Electrodeposited Sb Anodes through Solution Additives and Their Influence on Electrochemical Performance in Na-Ion Batteries."** Kelly recently obtained her Ph.D. at CSU in Materials Chemistry under the mentorship of Professor Amy Prieto.

In her interview below, she describes how the ARC's instrumentation and expertise played a critical role in enabling new insights into battery electrode materials.



SEM images of Sb thin films

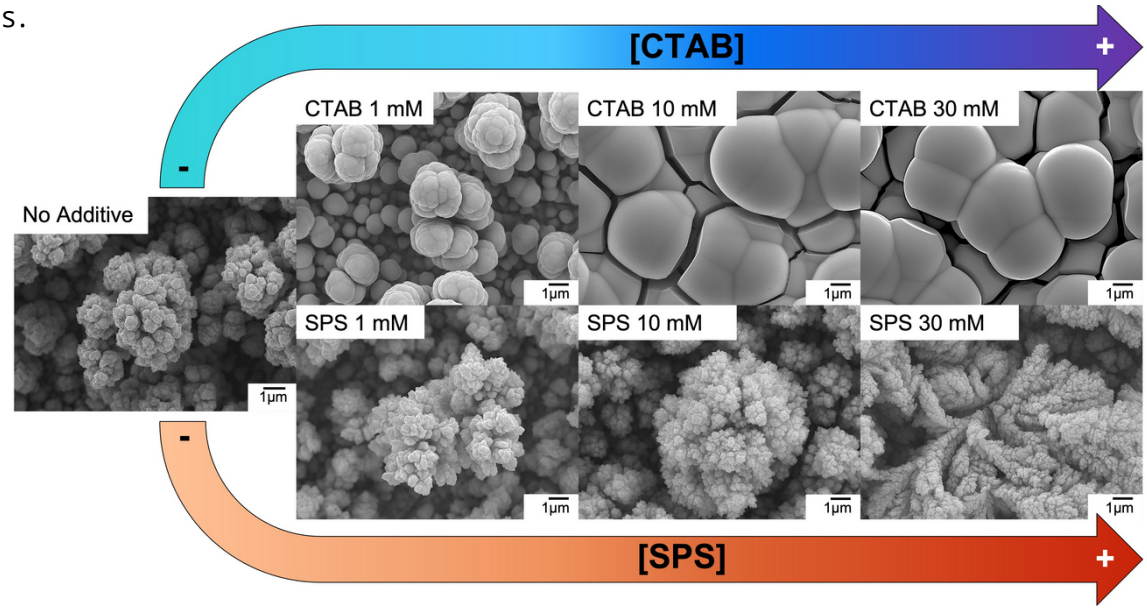
ARC TRIVIA

How much did the ARC spend on equipment service contracts and repairs this past year?

We had the incredible opportunity to interview Dr. Nieto and ask her a few questions about her research. Below is a Q&A that discusses her findings and how the ARC has helped contribute to her success!

Q: Describe briefly what your latest research publication was about and some of the key findings

A: Alloy-based materials such as antimony (Sb) are of interest for both Li/Na-ion batteries due to their high theoretical capacity and electronic conductivity. Of the various ways to fabricate Sb films (slurry casting, sputtering, etc.) one promising route is through electrodeposition. Electrodeposition is an industrially relevant synthetic technique that allows for the use of solution additives to control different characteristics such as film uniformity, morphology, and electrical conductivity. In this study, Sb films were electrodeposited from aqueous-based electrolyte baths with varied concentrations of cetyltrimethylammonium bromide (CTAB) and bis(3-sulfopropyl) disulfide (SPS). We report that these solution additives significantly influence the morphology and crystallinity of the deposited Sb film which consequently affected its performance as an anode for sodium-ion batteries. The addition of CTAB led to amorphous, strained films with smooth, densely packed particles and this is likely due to CTAB inhibiting the growth of Sb particles during the deposition. The SPS films are far more crystalline than their CTAB counterparts and exhibit columnar growth; we propose that crystallinity decreases with higher concentrations because of the accelerated rate of deposition induced by the additive. The combination of morphological and structural changes led to trade-offs in the cycle life and the rate of discharge of the Sb anodes. These studies provide valuable insight into the tunability of alloy-based films through electrodeposition and solution additives for battery applications.



Q: How did the resources and capabilities provided by the ARC contribute to the success of this paper?

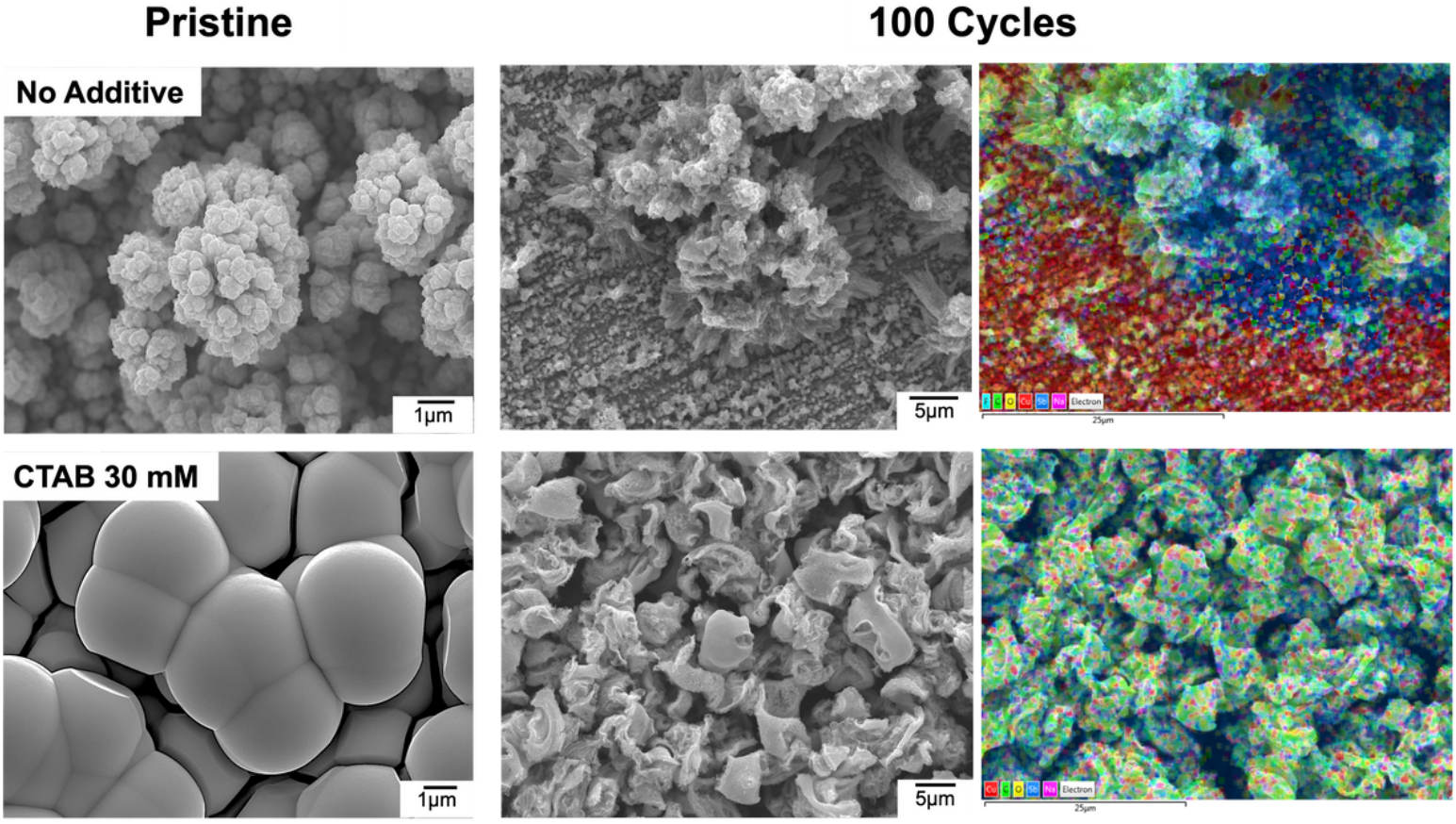
A: Without the resources provided by the ARC, this publication would not have been possible! By using the SEM, we were first able to find that slight changes in concentration of the additive/surfactant in solution could affect the electrodeposited Sb anode films. From this initial observation, we continued to probe how different additives could allow for control over morphology and crystallinity as well. By characterizing our Sb anode films with XRD, we found that the composition of the additive, in addition to its concentration, had a significant impact on the growth of these films and, therefore, the overall crystallinity.

Q: Can you share some specific examples of the advanced analytical techniques or equipment you utilized in the ARC that played a crucial role in your research findings?

A: In addition to characterizing the initial morphology of the film, we were able to perform ex-situ SEM and EDS on Sb films that had gone through 100 cycles of charging/discharging. Through this characterization technique, we identified that films deposited with CTAB had significantly improved adhesion to the copper foil substrate, as no copper was identified through EDS. Films deposited with SPS and without additives had poorer adhesion to the copper substrate and began to pulverize and delaminate from the substrate upon continuous cycling, leading to their shorter cycle life.

Q: In what ways has the availability of the ARC's services improved the overall quality of research projects in your group, and how do you envision its impact on future studies in your research field?

A: The resources provided by the ARC have enabled a plethora of research directions for our group to pursue, from electrochemically synthesizing and improving the properties/performance of anodes for both lithium- and sodium-ion batteries to understanding reaction mechanisms for the colloidal nanoparticle synthesis of photovoltaic materials. The services and expertise provided by the ARC and its staff are invaluable and will continue to help push our research forward in the field.



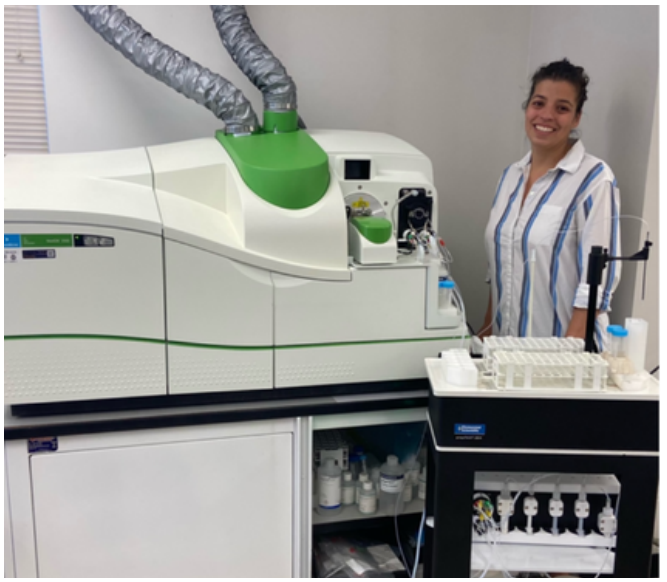
Thank you to Dr. Kelly Nieto for speaking to us about her research! We look forward to featuring more of our users' research. Want to have your research in the spotlight? Be sure to cite our RRID so that we can find you!

MEET THE INSTRUMENT AND EXPERT!

MEET OUR INDUCTIVELY COUPLED PLASMA MASS SPECTROMETER (ICP-MS)

ICP-MS (Inductively coupled plasma – mass spectrometry) is used to quantify elements in a sample. It is capable of monitoring and measuring most of the elements (stable isotopes) in the periodic table with the most notable exception of measuring H, C, N, and O.

ICP-MS can quantify total element composition or assess differences in isotope distribution of an element. Additionally, ICP-MS has low detection limits where many elements can be detected at low ppb to ppt levels. The ICP-MS is used in a wide range of research fields, with the major hurdle being sample preparation as ICP-MS can only measure elements in an aqueous acidic solution where nitric acid is typically used along with a microwave digestion system to digest samples to their elemental form. ARC has developed a method for quantifying a suite of elements of biological importance, but methods can be tailored to focus on the elements/ isotopes of interest.



MEET OUR ICP-MS EXPERT, DR. JACKIE CHAPARRO

Dr. Jacqueline Chaparro manages training, full service, maintenance, and education for the ICP-MS and microwave digestion system in the ARC for over eight years. Her duties are split between managing the ICP-MS in the ARC and managing Dr. Jessica Prenni's laboratory. She completed her undergraduate degree in chemistry at NYU, her MS in Forensic Science at John Jay College of Criminal Justice, and her Ph.D. in rhizosphere biology at CSU.

EQUIPTMENT UPDATES

L-BAND NOW OPERATIONAL ON THE EPR

We have installed the Cryostat (CF935) for the L-band bridge and resonator (ER 4118 split ring) in our EPR system which is available for 4-150 K measurements. The L-band (1–3 GHz) has lower frequencies which are preferable to ensure deeper penetration of electromagnetic waves. We can see higher order splitting in the L-band EPR spectrum related to small interactions. The low temperature enables better resolution and low concentration measurements. Please contact indrani.bhowmick@colostate.edu to learn more about the system.

TEAM UPDATES

Goodbye, Raj!

Congratulations to Dr. Prithwiraj De, who will be leaving ARC to take a position working in carbohydrate chemistry at CSU. We are grateful to him for his time with ARC and excited for him to be returning to the field of research that he came to us from! Best of luck to you, Raj!

THE ARC IS HIRING

We are thrilled to announce an opening for an X-ray Crystallography and Instrument Scientist to join our Team. This position will manage the ARC's X-ray diffraction laboratory, maintain instrumentation, oversee day-to-day operations, train and assist users, and perform detailed analyses on samples submitted to the facility, including small molecule single crystal structure solutions. If you are passionate about X-ray crystallography and have experience with XRD techniques and instrumentation, consider applying!

https://jobs.colostate.edu/postings/131812

FIND US ON LINKEDIN!

Follow us on LinkedIn for updates and to connect with the ARC! Follow the link below to access our page:

https://www.linkedin.com/company/analytical-resources-core/mycompany/?viewAsMember=true

ARC TRIVIA ANSWERS

July Bulletin: What is the most heavily used instrument in the ARC?
Answer: The Bruker US 400 MHz NMR in CHEMR (~240h/month)

New Trivia: How much did the ARC spend on equipment service contracts and repairs this past year?
Answer: $410,000

ARC FALL SEMINAR SERIES

We are excited to launch our upcoming Fall Seminar Series, where the lab managers of the ARC will present the technologies, analytical services, and educational resources each of their labs have to offer. Join us as we delve into a diverse array of scientific instruments and methodologies, enabling you to harness the full potential of our facilities.

SEPT 6

Overview of the ARC - Karolien Denef, ARC Director

From advanced spectroscopy to high-resolution imaging, the ARC provides critical infrastructure and expertise to enable research advancements at CSU. This session will provide an overview of the analytical services, expertise and educational resources offered by the ARC, and highlight how scientists can harness these resources to advance their research and address complex challenges across various disciplines.

OCT 4

Mass spectrometry-based metabolomics - Corey Broeckling, Director ARC-BIO

Metabolomics is a phenotyping approach with a focus on small molecules in biological, environmental, or ecological samples. This seminar will provide an overview of metabolomics approaches using mass spectrometry, and describe some resources withing ARC for performing metabolomics analysis.

Further details will be posted on our website in the coming days.
https://www.research.colostate.edu/arc/arc-seminar-series/

THANK YOU FOR CITING US USING OUR RRID!

Check out recent publications using the ARC:

Seitz VA, Chaparro JM, Schipanski ME, Wrighton KC, Prenni JE. Cover Crop Cultivar, Species, and Functional Diversity is Reflected in Variable Root Exudation Composition. Journal of Agricultural and Food Chemistry. 2023 Jul 21. https://pubs.acs.org/doi/10.1021/acs.jafc.3c02912

Hayes CB, Carter O, MacWilliams JR, Cranshaw W, Chaparro JM, Prenni JE, Nachappa P. Biology and management of hemp russet mite (Acari: Eriophyidae). Journal of Economic Entomology. 2023 Jul 14. https://doi.org/10.1093/jee/toad137

Tryner J, Quinn C, Molina Rueda E, Andales MJ, L'Orange C, Mehaffy J, Carter E, Volckens J. AirPen: A Wearable Monitor for Characterizing Exposures to Particulate Matter and Volatile Organic Compounds. Environmental Science & Technology. 2023 Jul 7. https://pubs.acs.org/doi/10.1021/acs.est.3c02238

Nieto K, Windsor DS, Kale AR, Gallawa JR, Medina DA, Prieto AL. Structural Control of Electrodeposited Sb Anodes through Solution Additives and Their Influence on Electrochemical Performance in Na-Ion Batteries. The Journal of Physical Chemistry C. 2023 Jun 26. https://pubs.acs.org/doi/10.1021/acs.jpcc.3c01086

Using the ARC?

Please cite or acknowledge us by our Research Resource ID

RRID: SCR_021758

in publications that include any data generated in or by our facility